

COMMISSION OF THE EUROPEAN COMMUNITIES

Brussels, 26.10.2005  
SEC(2005) 1399

PART II

**COMMISSION STAFF WORKING DOCUMENT**

**Monitoring of Pesticide Residues  
in Products of Plant Origin  
in the European Union, Norway, Iceland and Liechtenstein**

**2003**

**ANNEX 1**

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## 1. NATIONAL AUTHORITIES AND CONTACT POINTS FOR PESTICIDE RESIDUE MONITORING

Authority	Contact person	Contact point
<p><u>Belgium:</u></p> <p>Federal Agency for the Safety of the Food Chain WTC III Boulevard Simon Bolivar 1000 Bruxelles</p>	<p>Jean-François Schmit</p>	<p>Tel.: +32 2 208.47.69 Fax.: +32 2 208.47.93 e-mail : <a href="mailto:jean-francois.schmit@afsca.be">mailto:jean-francois.schmit@afsca.be</a></p>
<p><u>Denmark</u></p> <p>Head of Division Danish Institute for Food and Veterinary Research Department for Food Chemistry Mørkhøj Bygade 19 DK2860 Søborg Denmark</p>	<p>A. Büchert</p>	<p>Tel.: +45 72 34 74 61 Fax: +45 33 95 60 01</p> <p>e-mail: <a href="mailto:ab@dfvf.dk">mailto:ab@dfvf.dk</a></p>
<p><u>Germany:</u></p> <p>Dr. Wilhelm von der Hude, Referat 313 Bundesministerium für Verbraucherschutz, Ernährung und Landwirtschaft Rochusstraße 1 D-53123 BONN Germany</p>	<p>Dr. Wilhelm von der Hude,</p>	<p><a href="mailto:wilhelm.vonderhude@bmvel.bund.de">wilhelm.vonderhude@bmvel.bund.de</a></p> <p>Tel.: +49 1888-529-4659 Or: +49 1888-529-4661</p>
<p><u>HELLAS:</u></p> <p>Ministry of Rural Development and Food General Directorate of Plant Produce Directorate of Plant Produce Protection Department of Pesticides 150, Sygrou ave. 176 71 KALLITHEA HELLAS</p>	<p>Mr. Theofanis KAFRITSAS</p> <p>Dr Michael KASTANIAS</p> <p>Ms. Kalliopi KOKKINAKI</p>	<p>Tel: +30 210 9212092 Fax: +30 210 9212090 e-mail: <a href="mailto:kafr_th@otenet.gr">kafr_th@otenet.gr</a></p> <p>Tel: +30 210 9212164 Fax: +30 210 9212090 e-mail: <a href="mailto:farmaka2@otenet.gr">farmaka2@otenet.gr</a></p> <p>Tel: +30 210 9212164 Fax: +30 210 9212090 e-mail: <a href="mailto:farmaka2@otenet.gr">farmaka2@otenet.gr</a></p>
<p><u>Spain:</u></p> <p>Ministerio de Agricultura, Pesca y Alimentación Dirección General de Agricultura Subdirección General de Medios de Producción</p>	<p>Mr. A. Yague</p>	<p>Tel.: +34 91 3478273/75 Fax: +34 91 3478316 e-mail: <a href="mailto:mpaniagu@mapya.es">mpaniagu@mapya.es</a></p>

Authority	Contact person	Contact point
<p>Agrícolas Avda. Ciudad de Barcelona, 118-120 28007 Madrid</p>		
<p><u>France:</u></p> <p>DGCCRF-bureau C2 télédoc 051 59, boulevard Vincent Auriol 75703 Paris Cedex 13 France</p> <p><i>and:</i></p> <p>DGCCRF-bureau C2 télédoc 051 59, boulevard Vincent Auriol 75703 Paris Cedex 13 France</p>	<p>Mme Séverine Dubus</p>	<p>Tel: +33 1 4497 2468 Fax: +33 1 4497 2486</p> <p>e-mail: <a href="mailto:severine.dubus@dgccrf.finances.gouv.fr">mailto:severine.dubus@dgccrf.finances.gouv.fr</a></p> <p>e-mail: <a href="mailto:c2@dgccrf.finances.gouv.fr">mailto:c2@dgccrf.finances.gouv.fr</a></p>
<p><u>Ireland:</u></p> <p>The Pesticide Control Service Department of Agriculture, Food and Rural Development Abbotstown Castleknock Dublin 15</p>	<p>Dr. D. O'Sullivan</p>	<p>Tel.: +353 1 6072614 Fax: +353 1 8204260 e-mail: <a href="mailto:Dan.Osullivan@agriculture.irlgov.ie">Dan.Osullivan@agriculture.irlgov.ie</a></p>
<p><u>Italy:</u></p> <p>Ministero della Salute Direzione Generale della Sanità Veterinaria e degli Alimenti (D.G.S.V.A.) Ufficio V Piazzale Marconi 25 00144 Roma</p>	<p>Dr. Antonio Consolino</p>	<p>Tel: +39 06 5994 6844 Fax: +39 06 5994 6865 e-mail: <a href="mailto:a.consolino@sanita.it">a.consolino@sanita.it</a></p>
<p><u>Luxembourg:</u></p> <p>Laboratoire National de Santé BP. 1102, L-1011 Luxembourg</p>	<p>Dr. Carole Dauberschmidt</p>	<p>Tel. :+352 491191-394 Fax: +352 494 258 e-mail: <a href="mailto:Carole.Dauberschmidt@lns.etat.lu">Carole.Dauberschmidt@lns.etat.lu</a></p>
<p><u>Netherlands:</u></p> <p>Keuringsdienst van Waren (Inspectorate for Health Protection) Hoogte Kadijk 401 NL-1018 BK Amsterdam</p>	<p>H. A. van der Schee</p>	<p>Tel.: + 31205244702 Fax: +31 20 5244700 e-mail: <a href="mailto:Henk.van.der.Schee@VWA.NL">Henk.van.der.Schee@VWA.NL</a></p>
<p><u>Austria:</u></p> <p>Bundesministerium für Gesundheit und Frauen Abteilung. IV/B/10 Radetzkystraße 2</p>	<p>Dr. Sylvia Frischenschlager</p> <p>Dr. Roland Grossgut,</p>	<p>Tel.: +43/1/71100-4628 Fax.: +43/1/71100-4681 e-mail: <a href="mailto:sylvia.frischenschlager@bmgf.gv.at">sylvia.frischenschlager@bmgf.gv.at</a></p> <p>Tel.: +43 50555 25700</p>

Authority	Contact person	Contact point
A-1030 Wien	Risikobewertung, Österreichische Agentur für Gesundheit und Ernährungssicherheit GmbH, Spargelfeldstrasse 191, A-1226 Wien	Fax.: +43 50555 25802 e-mail: <a href="mailto:roland.grossgut@ages.at">roland.grossgut@ages.at</a>
<u>Portugal:</u>  Direcção-Geral de Protecção das Culturas Quinta do Marques PT-2780 Oeiras	J. R. Santos	Tel: +351 21 446 4000 +351 21 44 6 4025 Fax: +351 21 44 2 0616 e-mail: <a href="mailto:juliasantos@dgpc.min-agricultura.pt">juliasantos@dgpc.min-agricultura.pt</a>
<u>Finland :</u>  National Food Agency P.O. Box 28, FIN-00581 Helsinki	Arja Kaiponen	Tel. +358 9 3931 529 Fax: +358 9 3931 592 e-mail: <a href="mailto:arja.kaiponen@elintarvikevirasto.fi">arja.kaiponen@elintarvikevirasto.fi</a>
<u>Sweden:</u>  National Food Administration Box 622, S-751 26, Uppsala	A. Andersson	Tel.: +46 18 17 56 41 Fax: +46 18 17 53 53 E-mail: <a href="mailto:aran@slv.se">aran@slv.se</a>
<u>United Kingdom:</u>  Pesticides Safety Directorate Mallard House, Kings Pool 3 Peaseholme Green York YO1 7PX	Ms T. Ware  Ms H. Cooke	Tel.: +44 1904 455 751 Fax: +44 1904 455 733 e-mail: <a href="mailto:Tracey.Ware@psd.defra.gsi.gov.uk">Tracey.Ware@psd.defra.gsi.gov.uk</a>  Tel.: +44 1904 455 756 Fax: +44 1904 455 733 e-mail: <a href="mailto:helena.cooke@psd.defra.gsi.gov.uk">helena.cooke@psd.defra.gsi.gov.uk</a>
<u>Norway:</u>  The Norwegian Food Safety Authority P.O. Box 383 2381 Brumunddal Norway	Cécile Blom	Tel.:+47 23 21 68 00 Fax: +47 23 21 68 01 e-mail: <a href="mailto:ccblo@mattilsynet.no">ccblo@mattilsynet.no</a>
<u>Iceland:</u>  The Environment and Food Agency of Iceland Suðurlandsbraut 24, 108 Reykjavík Iceland	Dr.Grímur Ólafsson	Tel.: +354 591 2000 Fax: +354 591 2020  e-mail: <a href="mailto:grimur@ust.is">grimur@ust.is</a>
<u>Liechtenstein:</u>	Dr. P. Malin	Tel.: +423 236 7311

Authority	Contact person	Contact point
Amt für Lebensmittelkontrolle und Veterinärwesen Postplatz 2 Postfach 37 FL- 9494 Schaan		Fax: + 423 236 7310 e-mail: <a href="mailto:info@alkvw.llv.li">info@alkvw.llv.li</a>
<u>European Commission</u>  DG SANCO/ D3 Rue Froissart 101 B-1049 Bruxelles	P. Pitton	Tel.: +32 2 2987622 Fax: +32 2 2965963 e-mail: <a href="mailto:Patrizia.Pitton@cec.eu.int">Patrizia.Pitton@cec.eu.int</a>
<u>European Commission</u>  DG SANCO/ D3 Rue Froissart 101 B-1049 Bruxelles	B. Drukker	Tel.: +32 2 2965779 Fax: +32 2 2965963 e-mail: <a href="mailto:Bas.Drukker@cec.eu.int">Bas.Drukker@cec.eu.int</a>
<u>European Commission</u>  DG SANCO/ F4 Food and Veterinary Office Office 0/174 Grange Dunsany Co. Meath Ireland	P. Shanley	Tel.: +353 46 9061739 Fax: +353 46 9061864 e-mail: <a href="mailto:Peter.Shanley@cec.eu.int">Peter.Shanley@cec.eu.int</a>
<u>European Commission</u>  DG Joint Research Centre Institute for Reference Materials and Measurements Food Safety and Quality Unit Retieseweg B-2440 Geel Belgium	Dr. Christoph von Holst	Tel.: ++ 32 14 571 221 Fax: ++32 14 571 221 e-mail: <a href="mailto:christoph.von-holst@cec.eu.int">christoph.von-holst@cec.eu.int</a> <a href="mailto:christoph.von-holst@irmm.jrc.be">christoph.von-holst@irmm.jrc.be</a>

## 2. COUNTRY COMMENTS

### BELGIUM

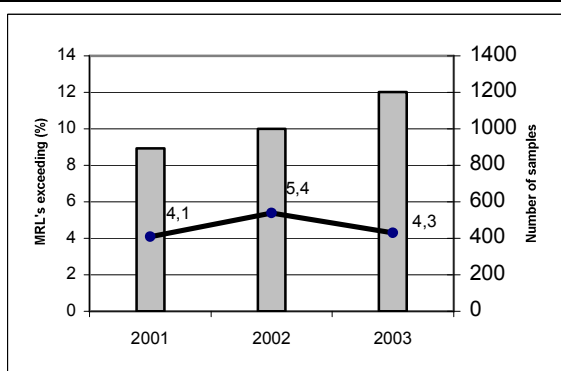
#### 1. SUMMARY OF RESULTS

In 2003, a total of 1291 samples of fruits, vegetables, cereals and processed products were taken on the Belgian market and analysed for the presence of pesticide residues (table 1). The monitoring included 341 samples of fruits, 859 samples of vegetables and 50 samples of cereals. In addition, 15 samples of baby food and 26 samples of other processed products were analysed. 66% of the samples of fruits and vegetables were of belgian origin, 29% were imported products and 5% were of unknown country origin.

National or EU harmonized Maximum Residue Level (MRL) were exceeded by 52 samples of fruits and vegetables (4,3%). 38% of these exceedances are related to imported products. Relatively high percentages (>10%) of MRL's exceedances were found for grapes, celery, lambs lettuce, peppers, mandarins and beans. Note that in this report exceedances were counted not taking into account the uncertainty on the analytical result. No exceedances were observed for cereals and processed products.

**Table 1:** Overview of the results of the pesticide residue monitoring programme in Belgium

Samples	analysed	without residues	with residues at or below MRL	with residues exceeding MRL	with residues exceeding MRL (%)	1.1.1.1. Products origin					
						Belgium		Other		Unknown	
						total	>MRL	total	>MRL	total	>MRL
Fruits	341	182	159	19	5,6 %	122	3	193	15	26	1
Vegetables	859	456	403	33	3,8 %	672	24	152	5	35	4
<b>Fruits &amp; vegetables</b>	<b>1200</b>	<b>638</b>	<b>562</b>	<b>52</b>	<b>4,3%</b>	<b>794</b>	<b>27</b>	<b>345</b>	<b>20</b>	<b>61</b>	<b>5</b>
Cereals	50	46	4	0	0 %						
Processed products	41	34	7	0	0 %						
<b>Total</b>	<b>1291</b>	<b>711</b>	<b>527</b>	<b>52</b>	<b>4,1 %</b>						



**Figure 1:** Comparison of the percentage of samples with residues exceeding MRL's in fruits and vegetables for the years 2001, 2002 and 2003.

For fruits and vegetables, the percentage of exceedances in 2003 (4,3%) is lower than in 2002 (5,4%). The number of samples analysed is noticeably higher than previous years. No residues were found in 53% of the samples of fruits and vegetables, 92% of the cereal samples and 83% of the samples of processed products.

Out of a list of 131 different pesticide residues sought in fruit and vegetables, a total of 47 were found at least once during the monitoring programme. The ten most frequently found pesticide residues, in decreasing order of frequency (found/sought) are: chlorpropham, prochloraz, bromide ion, chlormequat, imazalil, dithiocarbamates,

propamocarb, iprodione, cyprodinil and carbendazim. Most of these are found by single residue methods, which are only carried out when the presence of residues is expected (prochloraz and chlormequat for example). When counted in absolute number of findings, the ten most frequently

found pesticide residues, in decreasing order of number of findings, are: bromide ion, chlorpropham, iprodione, dithiocarbamates, propamocarb, tolclophos-methyl, procymidone, imazalil, chlormequat and carbendazim.

In cereals, out of 28 pesticide residues sought for, the following pesticide residues were found at least once : bromide ion, malathion and pirimiphos-methyl.

In processed products, only gamma-HCH was detected.

A maximum of 8 different pesticide residues were found in one sample of lettuce.

Four RAS-messages were issued in 2003 by the belgian food Agency for Indian grapes: 3 samples containing methomyl, and 1 sample containing both acephate and methamidophos. In all cases an exceedance of the acute reference dose was observed.

## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

- **Responsibilities:** The Federal Agency for the Safety of the Food Chain, under the responsibility of the Minister of Health, is the competent authority for the monitoring of pesticides residues in foodstuffs (<http://www.afsca.be>)
- **Drafting of the monitoring plan:** In selecting the commodities, the methods of analysis and the number of samples, several factors were taken into account: the average consumption, the Belgian production, the rate of exceedances and findings of previous years, the RASSF messages the analytical and budgetary possibilities and all other useful information. For less important commodities, a rolling programme is preferred. The different groups of fruits and vegetables were included as much as possible. The coordinated programme of the European Commission was of course included in the national programme.
- **Sampling:** *Sampling was done by trained officials according to directive 2002/63/EG, at auctions, importers, wholesalers, processors and exceptionally in retail.*
- **Measure taken:** In case of infringement, the responsible company/person is identified. When the exceedance of MRL is low, a warning is issued. When the exceedance is higher, an official report is made and sent to the responsible company/person and also to the legal service. The legal service proposes a fine. If the fine is not paid, or in case of repeated offences, the case goes to the court . When the dietary intake calculations indicate a risk for the consumer (evaluated following document SANCO/3346/2001) then a national and international rapid alert is issued and measures to protect consumers are taken (tracing and recall of the foodstuffs for destruction).

## 3. QUALITY INSURANCE

- The three officially recognized laboratories involved in the monitoring programme are accredited ISO 17025 for the most important analytical methods and commodities, and accreditation was gradually extended. Multiresidues methods and sometimes specific individual methods were performed on samples. All certificates of accreditation can be found on the website of the Belgian Organisation for Accreditation (<http://belac.be/>). The laboratories take into account EU Quality control procedures (ref. SANCO/3103/2000).
- Two laboratories participated in the European Commission's Proficiency Tests

## 4. OTHER INFORMATION

- The scope of the monitoring programme is to enforce the legislation. It does not provide for a total random analysis, as more samples are taken of food items expected to contain pesticides or to exceed MRLs. For example, pears were still followed closely, due to chlormequat problems in the past. Similarly, lettuce continues to be followed closely.



## DENMARK

### 1. SUMMARY OF RESULTS

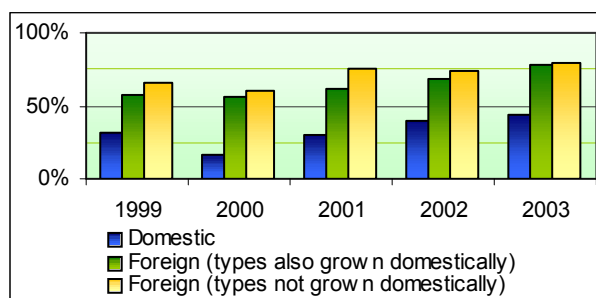
In 2003 a total of 1605 samples of fruit, vegetables and cereals (fresh, frozen or processed) were analysed: 443 samples of domestic origin and 1162 samples of foreign origin. The samples included 706 samples of fruit, 667 vegetables and 157 samples of cereals. In addition, 21 samples of baby food and 54 samples of other processed foods were analysed. Frozen food comprised 4% of the fruit and vegetable samples and 5% were organically produced. About 22% of the cereal samples were organically produced.

Residues exceeding the maximum residue levels were found in 45 samples (3%). Three of these samples were Danish produced, and 42 samples were of foreign origin. Most samples with exceedances were fruit and vegetables.

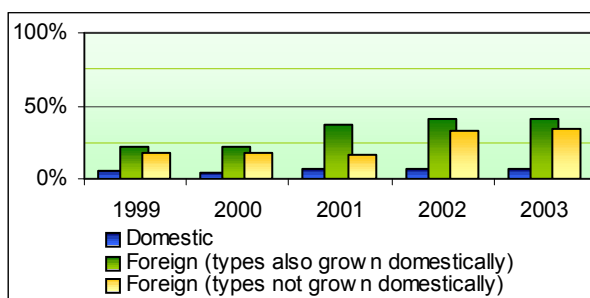
In non-organically grown foodcrops, pesticides were found on average in 49% of fruit and vegetables. Residues were found in 45% of Danish grown fruit, but in 80% of the samples of foreign origin. For vegetables, residues were found in 6% of the Danish samples and in 41% of the samples of foreign origin.

The residues found in cereals were mainly the growth regulator chlormequat (found in 25% of the samples). Multiple residues were found in 25% of the samples of fruit and vegetables.

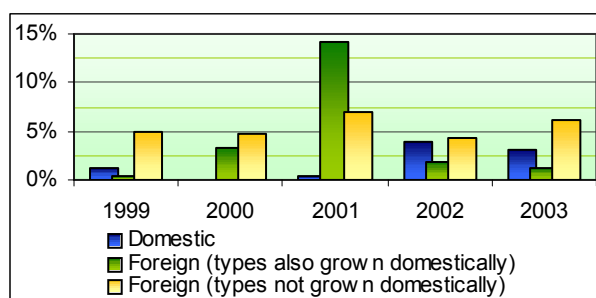
In organic food, residues of imazalil were found in one sample of banana from Peru, phosalon, pyrimethanil, tolylfluanid and trifloxystrobin in one sample of German apples and chlormequat in three samples of oat flakes of unknown foreign origin.



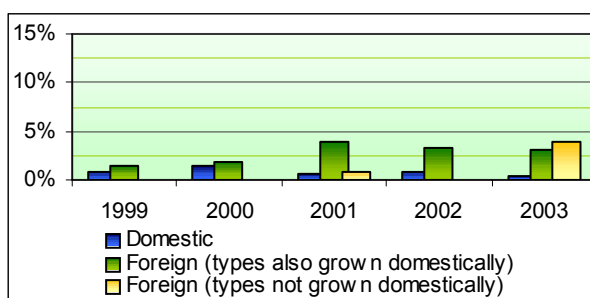
**Fruit** : Frequency of samples with pesticide residues



**Vegetables** : Frequency of samples with pesticide residues



**Fruit** : Frequency of samples with pesticide residues above the maximum residue limit



**Vegetables** : Frequency of samples with pesticide residues above the maximum residue limit

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

Pesticide monitoring in Denmark were in 2003 the responsibility of the Danish Veterinary and Food Administration under the Ministry of Food, Agriculture and Fisheries. The Institute of Food Safety and Nutrition designed the monitoring programmes and consolidated the collected data in cooperation with the Veterinary and Food Administration. The Regional Veterinary and Food Control Authorities were responsible for implementing the sampling plans and for enforcement actions.

Sampling plans were based on the dietary consumption pattern, production and import data, and monitoring results from previous years. All samples included in the centrally coordinated monitoring in 2003 were designed as surveillance sampling. A limited number of samples were analysed in regionally planned programmes. Results from these targeted samples have not been reported.

Samples were taken by authorised personnel from the 10 Danish Regional Veterinary and Food Control Authorities. Sampling was done mainly at wholesalers and importers; 2% of the samples were taken at food processing companies, 0.2% at shops and 0.7% of the samples at primary producers. Directive 2002/63/EC on sampling procedures for control of pesticide residues is implemented in Danish legislation.

Enforcement actions included warnings, fines and orders to improve internal control programmes.

### **3. QUALITY ASSURANCE**

The analytical methods are developed and validated by the Institute of Food Safety and Nutrition. The major part of the analytical work is performed at the Laboratory at the Regional Veterinary and Food Control in Copenhagen. The Institute of Food Safety and Nutrition performs confirmatory analysis and some special projects. Both laboratories are accredited for pesticide analysis in compliance with EN45001/ISO17025 by the Danish body of accreditation, DANAK (certificate numbers 315 and 350). The laboratories participated in the relevant FAPAS performance verification scheme and in the EU-proficiency test.

All samples of fruit and vegetables were analysed for at least 167 pesticides, isomers or metabolites. In addition, part of the samples was analysed for dithiocarbamates and imazalil. All samples of pears were analysed for chlormequat. All cereal samples were analysed for 92 pesticides, isomers or metabolites. Due to the methodology applied it is not possible to distinguish between dithiocarbamates included in the MRL definition and other dithiocarbamates for which no MRL has been fixed. Thus it is not possible to report confirmed exceedances for the maneb-group.

"Guidelines concerning Quality Control Procedures for Pesticide Residue Analysis" has been applied for all methods. Mass selective confirmation was performed for part of the GC multimethods and for the LC/MS-MS methods for cereals.

Analytical uncertainty is not applied in monitoring reports, but is always applied in case of enforcement actions.

### **4. OTHER INFORMATION**

Residues above the maximum residue limit, residues without maximum residue limits, samples with multiple residues and residues with acute reference doses were individually evaluated by toxicologists at the Institute of Food Safety and Nutrition.

Residues of pesticides with a low acute reference dose were detected in 14 samples of fruit and vegetables in quantities that could lead to an exceedance of the ARfD. One sample of table grapes from South Africa contained residues (0,16 mg/kg prothiofos) that were toxicologically unacceptable.

## GERMANY

### 1. Summary of the Results from the Federal Republic of Germany

The report contains the results of the tests carried out in fresh and frozen fruit, vegetables and cereals during the 2003 survey period. It includes all data transmitted by the laboratories of the official food control of the 16 Laender to the 'Federal Office for Consumer Protection and Food Safety, BVL'.

In 2003 in the Federal Republic of Germany a total of 10758 samples were tested for pesticide residues. In 4673 (43.4 %) samples no residues of pesticides could be quantified and in 5129 (47.7 %) samples, residues of pesticides were quantified in compliance with official, national and EU MRLs. 956 (8.9 %) samples contained residues of pesticides exceeding MRLs.

Table 1 shows the number of samples divided into domestic production and imports and takes into account the three Directives. The overview given in this table indicates that residues of imported samples exceeded MRLs more frequently than those of domestic foods. The reason could be seen in the fact that parts of the imported foods originated from countries with other climatic conditions which require an increased use of pesticides.

Table 1 Overview of the results of the tests performed on domestic and foreign produce.

Food item	Domestic samples				Import samples			
	total amount	without residues	residues up to MRL	residues above MRL	total amount	without residues	residues up to MRL	residues above MRL
Cereals	494	316	173	5	172	132	38	2
Fruit/vegetable	3660	1844	1601	215	6260	2228	3298	734
Processed products	35	30	5	0	1	1	0	0
Baby food	131	121	10	0	5	1	4	0
Total	4320	2311	1789	220	6438	2362	3340	736

The food samples were analysed for a total of 527 different pesticides from which 255 were detected at least in one sample. Residues of 141 individual pesticides exceeded MRLs.

### 2. Organisation of Monitoring Programmes and Sampling

In Germany there is a difference between Monitoring Programme and official food control. Parts of the data generated in both programmes correspond with the provisions of Directives 76/895/EEC, 86/362/EEC, 90/642/EEC and the Commission Recommendation of 19 August 2002 (2002/663/EC) concerning a coordinated Community monitoring programme. Therefore, the report includes the respective data from both programmes:

- Monitoring programme 1791 samples
- official food control 8967 samples.

Samples were taken on the level of producers, manufacturers, wholesalers, retailers, and restaurants according to a national sampling protocol published as official legal regulations. The active ingredients tested for were ingredients which had already been included in the Annexes to Directives 76/895/EEC, 86/362/EEC or 90/642/EEC.

The Federal Laender are responsible for the sampling, which is carried out by correspondingly trained official inspectors.

In the framework of official residue control, the samples are taken in a target-oriented manner in order to check if there are any infringements against food legislation (e.g. surpassing of MRLs). The kind and extend of the sampling are decided on by the Federal Laender. When infringements are stated, the Laender authorities apply adequate measures (follow-up examinations, fines and, if necessary, transfer of the case to public prosecution).

The monitoring is based on a representative collection of data. With regard to bio-statistical aspects, the samples are taken randomly according to defined sampling plans. The sampling plans to be used for this purpose are laid down every year by the Federal Government together with the Federal Laender.

### **3. Quality Assurance**

All the 35 laboratories transmitting data to the 'Federal Office for Consumer Protection and Food Safety, BVL' have been accredited and participated in proficiency tests.

Analytical methods used in the framework of food surveillance and of food monitoring comply with the requirements set out in the 'Guidance document on residue analytical methods' (SANCO/825/00), including the criterion that the average recovery rates must be in the range of 70-110% and the relative standard deviation  $\leq 20\%$ . If these requirements are met, measuring results are directly included in the assessment, without consideration of actual recovery rates and deviations. If the requirement is not fulfilled, specific rules may be agreed in justified cases.

The analytical uncertainty is only considered if measured residues indicate that maximum residue levels are exceeded. In this case, the measured value is in general reduced by twice the standard deviation based on the precision found in proficiency tests according to the Gilsbach method (1998) (1). With food of plant origin, the reduction is an over-all 60%, over all ranges of concentration, and with food of animal origin, the measurement is reduced by twice the theoretical standard deviation as derived from the Horwitz curve for the concentration range in question. It is taken for sure that maximum residue levels are exceeded when measured values, reduced by the respective deviation, are still above the respective limit value.

(1) Gilsbach,W.: Abschätzung der Messunsicherheit bei der Rückstandsanalytik von Pflanzenschutzmitteln. Lebensmittelchemie 52(3), 1998, pp. 95-96, and correction in 52(4), 1998, p. 133

## GREECE

### 1. SUMMARY OF RESULTS

A total number of 2082 samples for a total number of 108 pesticides were analysed by 8 laboratories, including 1620 samples of fresh and frozen fruit and vegetables, 35 samples of cereals and 427 samples of processed products.

➤ **fresh and frozen fruit and vegetables:** 1620 samples

1170 were domestic samples (72.2% of the total number of samples), while 450 samples (27.7%) were imported ones:

- 1241 samples (76.6%) contained not detectable residues of the pesticides sought.
- 342 samples (21 %) contained detectable residues at or below MRLs.
- In 37 samples (2.3%) residues of one or more pesticides at concentrations exceeding the EU-MRLs were detected.

From the above 1620 samples analyzed, 136 samples were also examined for the pesticides specified in the 2003/10/EC Commission Recommendation.

The total number of pesticides sought in fresh fruits and vegetables was 108. The 10 most frequently pesticides found in fresh fruits and vegetables were: maneb group (13,0%), aldicarb (5.5%), chlorpyrifos (5.2%), endosulfan (4.8%), procymidone (4.3%), benomyl group (4.1%), captan (3.3%), methamidophos (2.8%), phosalone (2.4%), iprodione (2.2%)

➤ **Cereals:** 35 samples

17 were domestic samples (48,6% of the total number of samples), while 18 samples (51,5%) were imported ones:

- 31 samples (88,6 %) contained not detectable residues of the pesticides sought whereas, 4 samples (11%) contained detectable residues at or below MRLs and none sample was found with residues of one or more pesticides at concentrations exceeding the EU-MRLs.

The total number of pesticides sought in cereals was 82. The 6 most frequently pesticides found in fresh fruits and vegetables were: dichlorvos (8.0%), chlorpyrifos (4.0%), deltamethrin (4.0%), dichloran, endosulfan and malathion (4.0%).

➤ **Processed products:** 427 samples

427 samples of virgin olive oil – all samples were domestic ones- were analysed:

- 224 samples (52.5 %) contained not detectable residues of the pesticides sought.
- 195 samples (46 %) contained detectable residues at or below the MRL.
- In 8 samples (1.9 %) residues of one or more pesticides at concentrations exceeding the EU MRLs were detected.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

#### • Responsibilities

The annual monitoring plan (including national and EU co-ordinated monitoring programme) was organised by the **central** competent authority. The responsibilities of each of the 8 laboratories involved regarding the number of samples of each commodity that should be analysed by each laboratory and the areas of sampling were well defined by this program. The responsible for the EU co-ordinated program laboratories as well as the responsible for the homogeneity exercise laboratory were also clearly stated.

#### • Design of Programmes (priorities, targeting)

The annual national monitoring plan takes into account the most important parameters such as: the number of samples for each commodity (depending on the produce, the cultivation area and the daily dietary intake contribution of each commodity), the sampling location and the personnel and analytical capacity of each laboratory.

- Sampling: personnel, procedures, sampling points

The responsible for sampling authorities, with the designated personnel, follow the methods of sampling (Commission Directive 79/700/EEC) as close as possible. Samples were taken from points of entry, wholesalers, retailers and farm gates.

- Enforcement action

4 follow-up enforcement samples were analysed, and were found without or with residues at or below LOD.

### 3. QUALITY ASSURANCE

- Status of accreditation of laboratories ; number of laboratories

From the 8 laboratories involved in the pesticide monitoring program 2003, the one has been accredited within 2002, whereas, the procedures for the accreditation of the rest 7 laboratories are still ongoing.

- Analytical methods used
- The Dutch Manual (5th edition 1988) for gas chromatographic analysis with NPD, ECD, TSD and PFPD
- Multiresidue method 5 for organophosphorus compounds, Ministry of Welfare, Netherlands, FIFTH EDITION
- The proposed EU method for dithiocarbamates
- A French UV method for benzimidazoles
- EPA Methods 5 and 531.1 and AOAC international protocol 29A05, described a direct inject method which employs gradient liquid chromatography with fluorescent detection, accomplished by post-column hydrolysis and derivatization of the eluted carbamates.
- For olive oil, a method developed in the one lab and published in Journal of Chromatography
- Participation in proficiency tests

From the 8 laboratories involved in the pesticide monitoring programme 2003, the one laboratory has participated to all 5 Proficiency Tests organised by the EU up to 2003, another in 2 Proficiency Tests (P.T.I, P.T II) organised by the EU, and a third one in both the 4<sup>th</sup> and 5<sup>th</sup> Proficiency Tests of EU, for 2002 and 2003 respectively.

The rest 4 laboratories, have participated to a proficiency test on pesticide residues for 11 Greek laboratories, organised in 2002.

- Implementation of EU quality control procedures

The EU quality control procedures are followed as close as possible. The screening of samples by GLC involves the use of matrix-matched standard mixtures containing the most commonly encountered pesticides, including at least two pesticides with a poor or variable GC response or recovery. Bracketing calibration is used. Although most quantitations are carried out using calibration curves with 3-5 levels covering a limited concentration range, a single calibration point is sometimes used, for example for confirmation of a residue, which has already been quantified on a different GC system. Routine recovery checks and blanks are carried out approximately every 20 samples rather than on the basis of batches, because of the uneven flow of samples to the laboratory.

### 4. OTHER INFORMATION

#### ➤ **Details of homogeneity exercise**

The homogeneity exercise was performed for the combinations peppers/endosulfan, peppers/chlorpyrifos, cucumbers/endosulfan and grapes/chlorpyrifos in samples taken from a single producer. This exercise was performed as recommended by 2003/10/EC Commission Recommendation.

#### ➤ **Details of risk assessment**

In the case of infringements, the evaluation of the acute risk is routinely carried out for those pesticides which have acute toxicity and where acute Reference Doses (acute RfDs) have been set. The acute risk is evaluated by using the UK Consumer Exposure Model.

## SPAIN

The Spanish Pesticide Monitoring Programme for pesticide residues in food was carried out by the Autonomies Communities coordinated by the Spanish Ministry of Agriculture, Fish and Food.

The objectives of the programme were to assume that:  
The pesticide was used accordingly with the Good Agriculture Practice.  
The health of consumer was protected.

The vegetables were collected by the inspectors following the requirements of the directive 2002/63/CE (Real Decreto 290/2003 de 7 de marzo) at the productions and wholesalers level because more that 95% of the Spanish food is produced in Spain a very few are imported food. Then they are sent to the laboratories. The monitoring programme has been co-ordinated by Ministry of Agriculture Fisheries and Food and included the European and national action.

### 1. SUMMARY OF RESULTS:

The total number of samples in Co-ordinate Programme and Spanish Programme 2003 were 3.648, and the repartition were 1.333 (36,51) fruits, 1.913 (52,43) vegetables and others vegetables, the 402 (11,01%) cereals.

For fruits, vegetables and others vegetables the samples that exceeded the 147 MLRs were about 4,8%. For cereals only 3 samples (0,7%), was above the LMRs.

The pesticides that were found the most frequently were according with the crop:

**Citrus fruits:** chlorpyrifos, dicofol, imazalil, malathion and methidathion.

**Pommes fruits:** captan+folpet, chlortalonil, diphenylamine, etoxiquin, imazalil, phosmet, procymidone

**Stone fruits:** acephate, fenthion, maneb group, phosmet.

**Grapes:** chlorpyrifos, fenitrothion, maneb group, procymidone.

**Strauwerry:** acrinatrin, chlortalonil

**Banana:** dicofol, dimethoathe.

**Tomato:** carbendazime, chlortalonil, chlorpyrifos, cypermethrine, endosulfan, procymidone

**Pepper:** cypermethrine, endosulfan, procymidone.

**Lettuce:** cypermethrine, imidachloprid, maneb group, procymidone.

Products	Sample Analysed	Sample not residues	Sample with residues at or below LMR	Samples >LMR	Samples >LMR EC	Samples >LMR N
<b>Fruits</b>	1.333	604	653	76	69	7
<b>Vegetables</b>	1.913	1.416	416	81	71	10
<b>Cereals</b>	402	382	17	3	3	0
<b>TOTAL</b>	3.648	2.402	1.086	160	143	17

## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING.

Coordinated responsibility: Sub. General de Medios de Produccion Agricolas (Dr. General de Agricultura) from Agriculture Ministry.

This programme takes into account:

- Proportion of the crops accordingly with the production.
- Requirements from EU co-ordinated programme
- Some special actions concerning problems with certain crop.

The responsibility personal are the inspector and analyse samples from the Comunidades Autonomas, then take samples according with the Directive 2002/63/EC, and they following the Manual Proceeding made at national level co-ordinated by S. G. Medios de Produccion Agricolas.

Samples normally are taken from wholesales, also are taken in farmgate and occasionally and retail. All samples are the national production taken into account more of 95% is for our consumption and not more that 5% are imported vegetables.

## 3. QUALITY ASSURANCE

14 laboratories carried out the monitory programme but until now their report is not yet accordingly with the European format that was make at Central Service.

Four laboratories, Madrid, Valencia, Almeria and Burgos were accredited by ENAC and others are in very advance phase to obtain the accreditation. The accreditation can be occurred only if methods was validated by collaborative study. The validation "in house" required a lot of investigation. For the new molecules the accreditation seems difficult to obtained. The four laboratories acredited were assuming approximately 40% of the samples.

12 laboratories to take part in Proficiency EUPT-5 from European Commission and all laboratories are participate in the Spanish proficiency Test-2003.

All samples were analysed by multiresidues method and same of there by simple method.

For multiresidues methods the samples were extracted by solvent (acetone or ethil acetate) then partitioned by liquid-liquid. The purification for organo-halogenated and organo phosphate molecules were performed by cartridge set-pack, florisil or GPC. ECD, NPD, FPD conduct the determination or hall detector GC/MS is used, as confirmation for samples above MRL's when the techniques is applicable.

Additional methods were used for determination of methyl carbamate, maneb groups, inorganic bromides, benomyl group and so on.

191 pesticides can be research and we have found 86 (45%)



## FRANCE

### • 1. SUMMARY OF RESULTS

The 2003 programme resulted in the analysing of 3375 samples of fruits, vegetables, cereals and processed products marketed in France, including 247 as part of targeted surveillance. The breakdown of samples according to origin was as follows: 68.7% were of French origin, 17.7% were of European Union origin and 13.6% were imported from third countries. The analyses detected and analysed 236 active substances. The most often found molecules were insecticides and fungicides.

Concerning only the **results of the fruits and vegetables monitoring plan**, 49.9% of the samples did not contain residues. Contamination levels under the maximum residue limit (MRL) were detected for 43.6% of the samples. 93.5% of the fruits and vegetables analysed therefore complied with regulations. MRLs were exceeded in 6.5% of cases.

**Among vegetables**, there was more than 10% of non-conformity for cucumbers (11 exceedances out of 85 samples), peppers (9 exceedances out of 92 samples), witloof (9 exceedances out of 79 samples), and lettuces (36 exceedances out of 307 samples). In contrast, there was less than 4% of non-conformity among cauliflowers (2 exceedances out of 69 samples), and potatoes (7 exceedances out of 217 samples). Globally 7% of vegetables were non-conforming and 59% did not contain residues.

**As for fruits**, exceedances mainly concerned citrus fruits like lemons (9 exceedances out of 79 samples), oranges (11 exceedances out of 116 samples), mandarins (7 exceedances out of 86 samples), as well as peaches (10 exceedances out of 101 samples), and strawberries (8 exceedances out of 93 samples). In contrast, pears and apples had few MRL exceedances—respectively 2 exceedances out of 90 samples and 3 exceedances out of 217 samples. Globally 5.5% of fruits (citrus fruits and exotic fruits included) were non-conforming and 35.5% did not contain residues. **Cereals and cereal products** did not contain residues in a quantity exceeding regulations in force (100% compliance).

23.7% of samples of fruits, vegetables, cereals and processed products analysed as part of the 2003 programme (follow up enforcement and surveillance samples) contained more than 2 residues at contents often under MRLs. This ‘multiple residues’ phenomenon concerned above all lettuces, citrus fruits, apples, strawberries, grapes and pears.

The inspections of organic produce are not reported in this document.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

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The monitoring programme for plant pesticide residues was carried out by the *Direction Générale de la Concurrence, de la Consommation et de la Répression des Fraudes* (DGCCRF—General Directorate for Competition Policy, Consumer Affairs and Fraud Control). The aims of this multi-year Community programme are to:

1. Monitor compliance with MRLs, and therefore compliance with reported good agricultural practices,
2. Collect data to assess actual dietary exposure of consumers to pesticide residues.

This programme takes into account:

- The results of the 2002 monitoring programme,
- The requirements of the European Union coordinated programme,
- The dietary proportion of plant products,
- The specific and sometimes targeted inspections of certain fruits and vegetables (specific exercise).

Each department received a crop sampling plan. Inspections were made at every marketing level, with adjustments. The inspection of cereals grains generally took place at the storage stage, at silos, or at the processing stage, at plants. Cereal products, fruits and vegetables were sampled at retailers or wholesalers. Samplings at growers were less frequent. For all products entering the French territory, specific action was deployed at points of arrival. A priori, domestic and imported production was not broken down, although a 70/30 ratio has prevailed for several years.

Inspectors are trained and carried out crop sampling by following the updated monitoring programme. In effect the quantities to be sampled have been changed recently to take account of changes at European and international level.

In 2003, the french targeted programme focused on lettuces and tropical root crops.

### **3. QUALITY ASSURANCE**

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Six DGCCRF laboratories took part in the monitoring programme and targeted surveillance programme. All the laboratories are COFRAC accredited (except for that in Bordeaux). The Strasbourg laboratory drew up the summary of the results.

The laboratories used multi-residue techniques and sometimes specific adapted methods. The techniques for multi-residue analyses used solvents for extraction (acetone or ethyl acetate), then liquid-liquid partition. Halogenated molecules were purified on adsorbents (Florisil or Bio-beads) or by gel permeation. Determination and quantification were performed by GLC or HPLC with adapted detectors such as ECD, FPD, NPD, Hall, MS, UV, and fluorimetry. GC/MS and HPLC/MS were used for confirmation most of the time. Specific methods were used for the determination of dithiocarbamates, bromides, maleic hydrazide, chlormequat and benzimidazoles and some other active substances.

The laboratories participated in proficiency tests organised by BIPEA, FAPAS, the European Union, and /or CHECK.

The “guidance concerning quality control procedures for pesticide residue analysis” was applied to most of the methods. Some difficulties arose during the application to special methods.

Measurement uncertainties were not taken into account.

## IRELAND

### 1. SUMMARY OF RESULTS

A total of 1022 samples of food of plant origin was sampled and analysed for pesticide residues in 2003. Of the samples analysed 986 were for the routine monitoring programme, including the EU co-ordinated plan, while the remaining 36 related to enforcement samples which were taken to deal with concerns arising with respect to the possibility of excessive pesticide residues being present in food.

As part of the surveillance work programme a total of 76 different fruit and vegetable commodities were analysed for residues of 89 different pesticides and their metabolites for the first 9 months of 2003 while a further 24 pesticides were added to this list from October to the end of 2003. In the case of fruit and vegetables 44% of the samples analysed were found to contain one or more pesticide residues and of those samples containing a pesticide residue 3.6% were found to contain a residue which exceeded a harmonised EU MRL. A range of cereal samples, including raw cereals and processed cereal products, were analysed for the same range of pesticides. The % of cereal samples containing residues was lower than for fruit and vegetables at 7.5% and only 1% of these cereal samples exceeded the MRL.

Dietary intake assessments were carried out in all cases where an MRL was exceeded. These assessments did not indicate that there was an unacceptable danger for consumers associated with these MRL exceedances.

The range of pesticides found in the samples analysed in 2003 are very similar to those found in previous years with 47 different pesticides being detected in fruit and vegetables. As expected a more limited number of pesticides, 5 in total, were detected in the cereal samples analysed and all appear to be present as a result of post harvest, in store, applications of insecticides. The most common pesticides found in fruit and vegetables correspond with those found in 2002. The most commonly found pesticides in fruit and vegetables for 2003 were the fungicides thiabendazole (11.5% of samples), carbendazim (9.6% of samples), captan (6.3% of samples) and iprodione (4.8% of samples) while the most common insecticide detected was chlorpyrifos (5.2% of samples).

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

The Irish monitoring programme was finalised following discussions between the Pesticide Control Authority of the Department of Agriculture and the Food Safety Authority of Ireland. The programme was agreed in November of 2002 between these two organisations and the programme was then implemented by the Pesticide Control Service.

The programme was designed by taking account of

- the current consumption patterns of Irish adults on data which was sourced from the 1996 to 1998 IUNA survey.
- historical monitoring data available with respect to the range of pesticides being found in food of plant origin.
- The co-ordinated EU monitoring programme for 2003.
- The manner in which food is handled prior to consumption. For example lettuce which is consumed in the raw state would be given a higher priority than a corresponding fruit or vegetable which was processed or cooked prior to consumption.

- the capacity of the laboratory to analyse samples.

Samples are taken in accordance with EU sampling Directive 2002/63. The sampling is carried out by two officers which work directly for the Pesticide Control Service and for which sampling of food of plant origin is their main responsibility. Sampling is normally taken at wholesale level but very occasionally it may be carried out at retail level.

Enforcement action is activated following an MRL breach or if information is transmitted to the Pesticide Control Authority to indicate that there may be excessive pesticide residues present in specified food of plant origin. An MRL is considered to be exceeded when the residue found is numerically greater than the indicated MRL. However enforcement action will not be taken unless the residue found is greater than the sum of MRL and the uncertainty of the analytical method.

### 3. QUALITY ASSURANCE

Monitoring laboratories: There is only one laboratory involved in the official control of pesticide residues in food of plant origin. This laboratory is the Pesticide Control Laboratory of the Department of Agriculture and Food and is accredited to ISO 17025 standard.

The analytical methods used for the analysis of pesticide residues are as follows

- Multi residue method 1, *Analytical Methods for Pesticide Residues in Foodstuffs*, 6<sup>th</sup> edition, 1996, General Inspectorate for Health Problems, Ministry of Public Health, Welfare and Sport, The Netherlands. The variation of the method uses ethyl acetate as the extraction solvent and is used for the analysis of residues of organophosphorous, organochlorine and benzimidazole pesticides in cereals.
- The Becker method, *A multi residue method for the simultaneous determination of plant protection chemicals in plant material*, Dtsch. Lebensm. Rundsch. 75, 148-152, 1979, using a gel permeation column instead of the silica gel/activated charcoal column specified.
- The method for the analysis of benzimidazole compounds in use is based on that developed by Hiemstra, M., J.A. Joosten and A. de Kok, J. AOAC Int. 78, 1267 -1274, 1995.

Proficiency tests: the Pesticide Control Laboratory participated in the 2003 EU proficiency study which are funded by the EU Commission and also participated in four FAPAS organised proficiency studies for pesticide residues in food of plant origin.

The laboratory incorporates the majority of the EU quality control procedures into its work procedures. Further details are provided in attached Table G.

Analytical uncertainty for 2003 was based on internal laboratory recovery studies. In future the laboratory will use the  $\pm 50\%$  generic figure, which was proposed at the EPRW 2004.

### 4. OTHER INFORMATION

The laboratory did not carry out any homogeneity exercise during 2003.

In all cases where an MRL is exceeded a risk assessment is carried out to determine its impact on consumers. A study is currently under way to determine the diet of Irish children. The survey element of this study will be completed by the end of 2004 and the study report will be finalised during 2005.

## ITALY

### 1. SUMMARY OF RESULTS

Of a total of 7688 samples (Tab.1 e 2), 2232 samples (29 %) with residues not exceeding permitted levels were found, while 126 (1,6 %) were found with residues exceeding permitted levels; no residues were detected in 5330 samples (69,4 %). The percentage of irregular samples is equal to 1,6% of which 1,8% for fruit and vegetables, 0,8% for processed products and 0 % for cereals.

The percentage of residues within legal limits is highest for fruit and vegetables (30,3 %), than for processed products (26,9 %) and cereals (9,5 %).

Cereals product samples analysed resulted regular.

SUMMARY OF DATA - YEAR 2003				
	Fruit and Vegetable	Cereals	Processed products	Total
Nr. Of samples	6,782	390	516	7,688
Regular samples	6,660	390	512	7562
Irregular samples	122	0	4	126
Irregular samples %	1.8	0	0.8	1.6

Tab. 1

PESTICIDE RESIDUES IN REGULAR SAMPLES				
	Fruit and Vegetable	Cereals	Processed products	Total
Nr. Of samples without residues	4.604	353	373	5.330
Nr. Of samples without residues %	67,9	90,5	72,3	69,4
Nr. Of samples with residues within legal limits	2056	37	139	2.232
Nr. Of samples with residues within legal limits %	30,3	9,5	26,9	29

Tab. 2

### 2. ORGANIZATION OF MONITORING PROGRAMMES AND SAMPLING

#### RESPONSIBILITIES

The Ministry of Health – Directorate General for Veterinary Health and Foodstuffs – coordinates and defines Italian official control programmes on foodstuffs, including the annual plans regarding pesticide residues.

#### STRUCTURE OF THE PLAN

The annual official control plans on residues of plant protection products are defined by Ministerial Decree 23 December 1992, transposing Directive 90/642/EEC, integrated by the Ministerial Decree 30 July 1993 regarding the programming of official controls for importation from Third Countries.

The National Program Pesticide Residues (P.N.R.A.) foresees a detailed programme implementing the checks to be carried out by the Regions and Autonomous Provinces of Trento and Bolzano, with indication of the minimum number and the typology of samples to be analysed. The division of the number of samples to be taken for each Region/Province is calculated according to the data on consumption and production of a given foodstuffs in the Region or autonomous Province concerned. The Decree contains some tables reporting the number of samples to be taken for each Region/Province for the following foodstuffs: vegetables, fruits, cereals, wine, oils, meat, milks and derivatives, eggs. The plan foresees also priority of a research of residues of plant protection products both in animal and vegetable origin foodstuffs.

As regards products of vegetable origin imported from Third Countries, the sampling is performed by Uffici di Sanità Marittima, Aerea e di confine terrestre of Ministry of Health, in at least 3% of a lot present at importation with a priority given to fruit and vegetable origin products.

### SAMPLING, STAFF, PROCEDURES AND SAMPLING SPOTS

Based on the programmes of the Regions and Autonomous Province, inspectors of a Local Health Units provides for implementation of sampling of foodstuffs to be tested for PPP residues.

The sampling spots indicated in P.N.R.A concerning products of plant origin are the collection centers and cooperatives for products coming from within the Region or Autonomous Province, specialised and non-specialised wholesale markets, wholesale stores, hypermarkets and supermarkets for products coming from outside the Region or Autonomous Province.

The sampling methods are those established by the Decree of the Ministry of Health of 23 July 2002, transposing Directive 2002/63/EC of 11 July 2002 regarding the methods of sampling for the Official control for pesticide residues in plant and animal origin products.

### MEASURES TAKEN

In case of irregular samples, the administrative or criminal sanctions foreseen by Law n° 283 of 30 April 1962 and Legislative Decree of 3 March 1993, n° 123 (transposing Directive 89/397/CE on official control of foodstuffs) are applied; contaminated foodstuffs are confiscated on a precautionary basis and/or destroyed.

## **3. QUALITY ASSURANCE**

### ACCREDITATION

Public laboratories, which have performed analysis for residues of plant protection products on foodstuffs according to the national monitoring programme, were 42. Various laboratories have already been accredited according to EN 45001, replaced by EN 17025, while other laboratories are in phase of accreditation. Implementation of analysis for the research of pesticide residues within Community coordinated programme was entrusted to 18 accredited laboratories located on the whole national territory.

### ANALYTIC METHODS

Analytic methods used mainly include GC multi-residue methods, associated with selective detectors (ECG, NPD, MS) and HPLC-UV.

### ATTENDENCE IN PROFICIENCY TESTS

Roughly 30 laboratories regularly take part to quality assurance tests organised by FAPAS, by the National Health Institute and by the APRA regional laboratories (Regional Agencies for Environment Protection).

These same laboratories attended the 5<sup>th</sup> Proficiency Test (year 2003) organised by “European Commission’s Proficiency Test on pesticide Residues in Fruit and Vegetable”.

Please find in Annex TABLE G LABORATORIES.

THE HEAD OF OFFICE V  
Dr. Antonio Consolino

Rome, 1/02/2005

## LUXEMBOURG

### 1. SUMMARY OF RESULTS

A total number of 107 samples were examined in the National laboratory of food control. 36% of the products (mainly wine grapes and wheat) were of Luxembourgian origin. In 2 samples oxadixyl levels higher than N-MRL were found. EC-MRLs were not exceeded. In 2 organic grown samples pesticide levels higher than 0.01 mg/kg were measured.

Table: Summary of the 2003 monitoring programme (co-ordinated and national programme)

Number of samples analysed (fruits, vegetables and cereals)	No of samples without residues	No of samples with residues at or below the MRL	No of samples exceeding EC-MRL	No of samples exceeding N-MRL
107	54	51	0	2

Dithiocarbametes (maneb group) were found most frequently (in 22% of the samples) followed by folpet (8%). The occurrence of these 'top two' pesticides however depended on the high sensibility of the dithiocarbamate method as well as on the commodity 'wine grapes' (folpet). Carbendazim (benomyl group) was frequently found in wine grapes and wheat, captan and vinclozolin in peas, respectively. These findings, however, do not appear in the report, as the concentrations of these pesticides were below the limit of quantification.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

Pesticide residues were analysed by a technician of the laboratory of food control (being part of the Laboratory of National Health, Ministry of Health). He was attended by a scientist who did not carry out routine analysis herself, however.

Due to these small capacities, the annual programme consisted mainly of the EC programme, the national programme consisted of herbal tea and strawberry samples.

The samples were collected by a food inspector of the food control unit. Imported products were sampled at wholesaler distribution points and retailers, local products were sampled at the central market in the City of Luxembourg and directly at local growers. Only routine sampling (no follow-up enforcement sampling) was done. As far as practicable sampling was done according to directive 63/2002/EEC.

### 3. QUALITY ASSURANCE

The laboratory of food control is the only laboratory doing pesticide analysis in Luxembourg. It was accredited in May, 2003.

2003, the laboratory participated in five proficiency tests concerning pesticide residues.

CS<sub>2</sub> (from dithiocarbamates, maneb group) were obtained by the Keppel-method and detected at very low concentrations by GC/MSD.

The benomyl group was detected with HPLC-UV as well as aldicarb, methiocarb, methomyl and thiram. For the later ones the method was not very sensitive, however. All the other pesticides were analysed by a gas chromatographic multi-residue method with ECD, NPD and MSD (DFG S19, modified). With this method, the recovery of lambda-cyhalothrin did not meet the analytical quality criteria.

A relative standard deviation of 30% for the analysis was taken when deciding whether a MRL was exceeded or not.

# NETHERLANDS

## 1. SUMMARY OF RESULTS

In 2003 a slightly lower percentage of non-compliances has been found for non-domestic samples than in previous year (Figure 1). For non-domestic products no products in special are responsible for the change. The slight rise in exceedances for Dutch products stems from strawberry, bean and carrot. Table 1 gives the most frequently non-complying pesticide/crop combinations with the main countries of origin. The Netherlands issued eight rapid or information alerts on pesticide residues. Table 2 gives an overview. For a number of grape samples, a product in the co-ordinated program, a homogeneity exercise has been performed. Table 3 summarises the results.

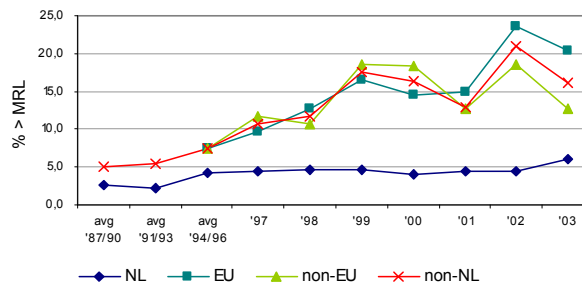


Figure 1. Percentage of MRL violations not including incidents

Table 1. Main products with high percentages of non-compliances, with corresponding pesticides and countries of origin.

Product	Pesticides	%>MRL	Countries
Grape *	methomyl, pyrimethanil, dimethomorph, acephate	26,4	India, Italy, Greece
Peach/nectarine	phosmet, etofenprox	17,9	Spain
Strawberry*	fludioxonil, endosulfan	34,4	Spain
Lettuce	dimethoate, pencycuron, oxadixyl	15,0	The Netherlands, France
Pepper (chili)	methamidophos, prothiofos, profenofos	36,7	Thailand
Bean	dimethoate	18,5	Kenya, Surinam

\*Taking into account an import tolerance decision for fludioxonil of 21-3-2003.

Table 2. Alerts to the RASFF system issued by the Netherlands.

Product	Pesticide	Country	Number
Grape	methomyl, monocrotophos	India	9
Lettuce	oxydemeton-methyl	Germany	1
Grape	methomyl	Greece	1
Long bean	monocrotophos	Surinam	1
Okra	monocrotophos	Surinam	1
Grape	parathion-methyl	Italy	2

Table 3. Summary of homogeneity factors (maximum unit concentration divided by sample mean) from exercise on grapes.

Pesticide	samples	mean	minimum	maximum
acephate *	2	6,5	2,3	10,7
carbendazim *	2	5,6	3,3	7,9
chloorpyriphos	2	6,0	3,7	8,3
ethiofencarb *	1	1,9		
iprodione	2	5,6	5,0	6,3
lambda-cyhalothrin	1	3,8		
methamidophos *	2	3,8	2,1	5,4
methiocarb *	1	2,2		
methomyl	3	4,3	3,4	5,4
monocrotophos *	1	6,5		
parathionmethyl	1	4,9		
quinoxifen	1	2,1		
spiroxamine *	1	3,5		
thiodicarb *	1	8,4		
vinchlozolin	1	2,6		

\* concentration in composite sample below reporting limit



## 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

The Food and Consumer Product Safety Authority performs the official monitoring in the Netherlands. The samples are taken without prior information about the presence of pesticides in the sample. Therefore, they represent the situation on the market for the product at that time. However, sampling is directed relatively more to products that need attention because of the violation rate in previous years. Therefore, high violation rates can indicate both an efficient sampling strategy and problems in the agricultural practice.

The monitoring program is primarily directed to major products in the consumption pattern, but some capacity is reserved to minor products. In the monitoring program special attention was given to chlormequat on pears, because of the high level of MRL-exceedances in 1999.

The Dutch Food and Commodity Law regulates the sampling procedure, i.e. the number of subsamples taken from a lot. This regulation is the implementation of the EC-directive 2002/63/EC. Inspectors of the five regional inspectorates are taking samples.

The main sampling points are the premises of the auction system for Dutch products and importers, warehouses and distribution centres of retail chains for both domestic and non-domestic products. At those inspection points it is clear who is responsible for the product, so that appropriate legal action can be taken in case of non-compliance.

## 3. ANALYSIS AND QUALITY ASSURANCE

One regional inspectorate (Northwest in Amsterdam) performs the analyses of the samples taken by all five regional inspectorates.

The general strategy is detecting as many pesticides as possible in one analysis by using Multi-Residue-Methods (MRMs). The Dutch method consists of an acetone extraction and a partition step of the residues into dichloromethane/petroleum ether followed by a chromatographic separation and selective detection of residues. The main detection method is gas chromatography (GC) followed by Ion-Trap Mass Spectrometric Detection (ITD). Only for some analytes not detectable sensitively enough by ITD, additionally GC with Electron Capture Detection (ECD) and GC with Nitrogen/Phosphorus (NPD) or Phosphorus/Sulphur detection (FPD) is used.

For pesticides not amenable to GC, High Performance Liquid Chromatography (HPLC) Multi- or Single Residue Methods are used:

- with UV-detection for the detection of fungicides thiabendazole, carbendazim, prochloraz and imazalil
- with post-column derivatisation and fluorimetric detection of N-methylcarbamates
- with ms/ms detection for chlormequat and propamocarb.

From September 2003 all sample extracts have been analysed by LC-MS/MS as well.

Dithiocarbamates are analysed as CS<sub>2</sub> using GC-ECD after decomposing with acidic tin-chloride solution and extraction into iso-octane.

Together these methods detect about 400 analytes.

The validity of the analytical results is governed by a quality assurance system complying with ISO17025. The multi-residue methods are within the scope of the accreditation of the laboratory.

The centralised laboratory has implemented the EU Guideline on Quality Control Procedures. It takes part in FAPAS and EU proficiency tests.

Only in case an MRL is exceeded by more than the estimated average measurement uncertainty of 30 %, legal action is taken.

## **AUSTRIA**

### **1. SUMMARY OF RESULTS**

In 2003 a total of 1491 samples were analysed under the co-ordinated program, the national pesticide monitoring program and as routine samples. Most of the samples were fresh fruits and vegetables (1322 samples). Beside that other products like cereals, processed products and baby food were analyzed.

44,9 % of all samples of fruits and vegetables were from Austria, 34,3 % from the European market and 20,3 % from third countries. For cereals this rates were 81,7 , 14,6 % and 3,7 % respectively. The rates for processed food were 59,8 %, 25,6 % and 4,9 %. Baby food was predominantly from the European market (92,3 %).

In 67,2 % of the samples of fruits and vegetables no pesticide-residues could be detected. 28,6 % of the samples had residues under the harmonized and/or national Maximum Residue Limits (MRL). In sum 95,8 % of the samples were in compliance with the regulations.

4,2 % of the samples of fruits and vegetables contained one or more pesticide(s) above the MRL.

In 189 samples (12,7 %) more than one pesticide was analysed. Up to 8 pesticides were found in some samples. The samples with more than 7 pesticides were in all cases grapes or peppers (8 samples).

In the samples were analysed up to 217 different pesticides. Totally 257 different pesticides were sought, of which 85 (33,1 %) were found. The most frequently found residues in fruits and vegetables were in the co-ordinated and national programme and routine samples: Maneb-group, Fludioxinil, Procymidone, Cyprodinil, Iprodione, Chlorpyrifos, Azoxystrobin, Endosulfane, Methomyl and Cypermethrin.

### **2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING**

The national pesticide monitoring is done according to a nation-wide sampling plan designed by the Institute of Applied Statistics and System Analysis (Joanneum Research, Graz) in co-operation with the Federal Minister of Health and Women. The plan was based on data concerning dietary consumption, production and import of fruits and vegetables and results of former measurements. Furthermore the results of earlier monitoring-programs, the analytical possibilities and the budgetary situation were taken into account, too. The co-ordinated programme of the European Commission was of course also done.

The samples were taken by trained officials from the local Food Inspection Service („Lebensmittelaufsicht“).

### **3. QUALITY ASSURANCE**

The analysis of the co-ordinated programme, the national monitoring programme and routine samples also were made by two laboratories for food control (Austrian Agency for Health and Food Safety, Institute for Food Control, Vienna and Institute for Food Control, Innsbruck). Two additional laboratories for food control of the Austrian Agency for Health and Food Safety (Institute for Food Control, Graz and Institute for Food Control, Linz) made the analyses of some samples of the national monitoring programme and routine samples. One Laboratory in Vienna (Regional Institute for Food Control in Vienna) analysed routine samples

The analytical methods were adopted from published methods of the Dutch federal laboratories („Analytical Methods for Pesticide Residues in Foodstuffs“, 6th Ed., General Inspectorate for Health Protection, Ministry of Public Health, Welfare and Sport, The Netherlands) and validated in the laboratories. The fruits and vegetables were analysed up to a maximum of 217 pesticides. The methods used were a GC multimethod with ECD-, NPD- and FPD-detection. GC/MS-methods are

primarily applied for confirmation purposes of the other GC methods, a HPLC-method for benzimidazol-fungicides and a spectrophotometric method for the sum of dithiocarbamates.

All laboratories involved in the co-ordinated programme and the national monitoring programme including the routine samples got the accreditation in the year 1998.

In 2003 three of the laboratories participated at proficiency tests, eg. the European Proficiency Test 5.

#### **4. OTHER INFORMATION**

Due to the fact, that there were some commodities for the national programme isolated, of which higher risk for residues was identified in the last years, these specific data are representative for the Austrian market, but the monitoring has to be seen partially as „targeted monitoring“. It was the aim, to reflect to the results of the last years and to choose special commodities of interest for further examination. This type of monitoring is foreseen for the next years.

Furthermore the routine sampling includes special samples, of which European alerts were given, too and thus the number of exceedance is higher than by doing statistical based sampling over all commodities and time of the year.

## **PORTUGAL**

### **1. SUMMARY OF RESULTS**

In 2003, a total of 297 surveillance samples of fruits and vegetables, including potatoes, were analysed for residues of 129 pesticides and 2 groups (benomyl and maneb), amounting to 153 analytes, although not all samples were analysed for all analytes.

The number of follow up enforcement samples was merely three, and only in one case the violative residue was present (dicofol in strawberries). About 50% of the samples of fruits and vegetables were analysed in the framework of the EU co-ordinated monitoring programme. Residues of at least one of the pesticides sought were found in about 41% of the sampling and multiple residues occurred in 42 fruits and vegetables (about 14% of the sampling). In total, 33 different pesticides have been found. The five most frequent pesticides detected were all fungicides (maneb group, iprodione, procymidone, dichlofluanid and benomyl group). The 6<sup>th</sup>, 7<sup>th</sup> and 8<sup>th</sup> most frequent ones were the insecticides methiocarb, chlorpyrifos, endosulfan. Azoxystrobin and dicofol were the subsequent most frequent compounds with the same rate of occurrence. Infringements to EC MRLs were found in 25 samples (8,4%) and 8 additional samples exceeded national MRLs, corresponding to about 11% violative samples in total.

The situation which most contributed to exceedence to national MRLs was the presence of methiocarb in aubergines from Spain. This resulted in aubergines being the commodity with the higher rate of infringements, although it should be noted that the MRL for methiocarb is set at the limit of quantification (0,05 mg/kg).

Lettuce and spinach showed again to be critical commodities in regard to infringements. The compound responsible for the majority of violations in these two crops together was the maneb group. Also strawberries showed a high rate of infringements due to the presence of benomyl (group) or dicofol, which use is no longer authorized in this crop.

Monitoring of cereals involved 63 surveillance samples which were analysed for about the same compounds as fruits and vegetables. Residues of at least one of the pesticides sought were found in 19 samples (about 30%) and multiple residues occurred in 5 samples. The only compounds found in cereals were malathion, pirimiphos-methyl, dichlorvos, deltamethrin and chlorpyrifos-methyl and no exceedences to EC or national MRLs have been found.

Analyses of processed food (other than baby food) involved 49 samples, which consisted mostly of wines. Residues of three different pesticides, at negligible levels, were detected in 5 samples of wine.

### **2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING**

The responsibility of monitoring programmes and sampling lies in the Ministry of Agriculture, except for baby foods, for which the Ministry of Health is responsible. For products of vegetable origin the responsible bodies are Directorate General of Food Quality Control and Inspection (DGFCQA) and Directorate General for Crop protection (DGPC), with the co-operation of regional agricultural services. Sampling is carried out by DGFCQA inspectors and by regional inspectors over the country, under DGFCQA co-ordination. In the autonomic region of Madeira sampling is the responsibility of the Regional Inspectorate of Economical Activities (IRAE).

Results produced by the laboratories are reported to DGFCQA and, in case of violations, risk assessment is carried out by DGPC. DGFCQA is also the body responsible for enforcement actions, such as official warnings, levying of fines or preparation of prosecutions by the court, according to the severity of infringements. In Madeira IRAE is responsible for these actions.

Monitoring programmes are elaborated in a meeting with the participation of representatives of the intervening bodies (sampling inspectors and analysts). The national programme for 2003 was based on the EU coordinated monitoring programme, which was extended to other pesticides according to

the capabilities of the laboratories. Strawberries, lettuce and spinach were summed up to the programme due to results of infringement found in previous years.

Sampling was carried out by trained officers, according to the procedures laid down in directive 2002/63/EC.

In the mainland samples were taken mostly at wholesale commerce and wholesaler's warehouses. Domestic cereals were generally taken in processing plants. In Madeira Island most samples were taken at retail.

### **3. QUALITY ASSURANCE**

The following pesticide residue laboratories contributed to the national monitoring programme: DGPC (lab 1) Regional Agricultural Directorate of Madeira (lab 2) and Regional Agricultural Directorate of Entre Douro e Minho (lab 3). None of the laboratories is accredited yet. Work for accreditation is ongoing in all laboratories, specially in lab 1 where more than 80% of quality and technical SOPs concerning the GC and the N-methylcarbamates method were implemented by the end of the year. EU quality control procedures are implemented in the 3 laboratories at variable degrees, as shown in Table G.

The GC multi-residue methods used are method P (lab1) and method M (lab2) in European Standard 12393. The determination of maneb group is based in European Standards 12396-1 (labs 1 and 3) and 12396-2 (lab 2). The benomyl group is determined by HPLC after ethyl acetate extraction and acid-base partition (lab 1 and 2). N-methylcarbamates are determined by HPLC-FLD with on-line OPA derivatization, after extraction and clean-up identical to method P above (lab 1) or after extraction and clean-up identical to method M above (lab 2). Organophosphorus insecticides precursors of sulphoxides and sulphones are analysed by oxidation of the cleaned extract obtained according to method P (lab 1). In 2003 labs 1 and 2 have both participated in the 5th EU proficiency test. Lab 1 also participated in one FAPAS exercise (MRM).

All values detected above MRL (mean of at least 2 separate analyses) are reported as infringements. However, administrative actions are taken only in those cases where the levels found exceed 1,4 x MRL. For values up to 1,4 x MRL action taken consists of official warnings. This figure is based on the estimation of a maximum standard uncertainty of 20% for a number of pesticides and application of a coverage factor of 2 for a confidence level of 95%.

### **4. OTHER INFORMATION**

Grapes were the commodity selected for duplicate sampling with a view to the homogeneity exercise. Results in tables F show homogeneity factors ranging from less than 2 to around 3.

The assessment of acute exposure for one violation of pirimicarb in lettuce and one violation of dicofol in strawberries (surveillance sampling) showed that the respective acute reference doses were exceeded. In both cases a health risk could not be excluded, specially for vulnerable groups. A follow-up enforcement sample confirmed the dicofol exceedence and led to destroying the remaining of the lot. Follow up enforcement sampling of the lettuce exceeding pirimicarb was attempted but the product was no longer on the market.

## **FINLAND**

### **1. SUMMARY OF RESULTS**

The total number of samples was 2158, including 1616 samples of fresh and frozen fruit and vegetables, 109 samples of cereals, 51 samples of baby foods and 382 samples of other processed foods. The share of third country samples was 54 % (1161 samples). The number of domestic and other EU member state samples was 420 and 577 respectively. The total number of samples includes 80 enforcement samples collected as follow-up of violations.

Pesticide residues above the reporting limits were found in 46 % of the fruit and vegetable samples, in 36 % of cereal samples, and in 16 % of processed products. Only two baby food samples (3.9 %) had detectable residues.

The frequency of pesticide findings was 18 % in domestic samples, 47 % in samples from other EU member states, and 49 % in third country samples.

The number of samples with residues exceeding the maximum residue levels (MRL) was 126 (5.8 %), out of them 102 were samples from third countries and 24 from other member states. All domestic samples complied with the MRLs. The samples with excess residues originated from 16 different countries. Analytical uncertainty was not taken into account when reporting the results. However, legal actions were taken only in case the measured residue minus analytical uncertainty was higher than the MRL.

In fruit and vegetables, residues of 81 different pesticides were found, and MRLs of 39 pesticides were exceeded. In cereals, residues of only six different pesticides were found. Pesticide residues were found in one out of 124 samples of organic products. The number of baby food samples was 51, and two samples had a residue above 0.01 mg/kg. Two RASFF notifications were sent during the year.

### **2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING**

National Food Agency (NFA) is the central competent authority responsible for directing, planning and developing food control in Finland. Municipal food control authorities perform the practical control. The control of imported food products and transit goods is assigned to the Customs administration.

The annual national sampling plan for control of pesticide residues is planned in co-ordination under the NFA. The sampling plan includes the national and co-ordinated EU-programmes. The sampling plan is designed to cover all important commodities of the diet. Priorities are set according to the consumption figures and known residue problems. Local health inspectors collect domestic samples from farms or retail shops, and Customs inspectors collect samples of imported foods from wholesalers. The sampling method of directive 2002/63/EEC was followed.

After each confirmed exceeding of MRL, either a legal action or a voluntary action by the operator was taken. In case of surveillance samples, the holder of the product was ordered to withdraw the remaining parts (if any left) of the inspected lot. Follow-up samples were taken from the next lots of the same origin. In case of enforcement sampling, the non-complying lot shall be destroyed, or in some situations, the lot may be processed to comply with the MRL (e.g. aeration to decrease fumigant residues).

### **3. QUALITY ASSURANCE**

The analyses were carried out in two accredited laboratories: Finnish Customs Laboratory (87 % of the samples) and Environmental Laboratory of the City of Helsinki (13 % of the samples).

Most analyses have been made by the gas chromatographic multiresidue method, which method is accredited in both laboratories. The number of pesticides monitored for in the two laboratories by the multiresidue method was 166 and 72 respectively. In addition, maneb group pesticides, inorganic bromide, hydrogen phosphide, maleic hydrazide, glyphosate, N-methyl carbamates, chlormequat and diquat were analysed in Customs Laboratory from selected samples.

Both laboratories have participated in the EU-PT5 proficiency test, and Customs Laboratory also in the FAPAS proficiency test (series 19, round 32, pesticides in baby food) and in the ring tests for LCMS measurements of pesticide residues organised by the National Food Administration of Sweden.

The quality control procedures as described in Commission Recommendation 1999/333/EC have been implemented in both laboratories but some elements only partly.

### **4. OTHER INFORMATION**

Sixteen homogeneity experiments were carried out. In six cases the grower code could be identified by the sampling officer and the samples were presumed to originate from a single producer. The other samples were presumed to be from mixed lots.

The homogeneity factors were as follows: endosulfan in peppers 3.1 (mean of five experiments), imidacloprid in peppers 2.4 (mean of two experiments), pirimiphos-methyl in peppers 6.9, endosulfan in cucumber 2.3, chlorpyrifos in grapes 2.6 (mean of three experiments), chlorpyrifos-methyl in grapes 3.1, endosulfan in grapes 1.9, methiocarb in grapes 1.3 and parathion-methyl in grapes 1.5.

The MRLs are published in the Decision 896/1999 of the Ministry of Trade and Industry (MTI). The decision integrates all Community MRLs and some old national MRLs. Since 1999, EC MRLs have been implemented by individual Decisions and Regulations of the MTI. Provisional MRLs or other national MRLs are not set. The MRLs of processed foods are derived from the MRLs of the fresh products taking into account the concentration or dilution caused by the process.

## SWEDEN

### 1. SUMMARY OF RESULTS

In 2003, a total of 2 352 surveillance samples of fruits, vegetables, juices, fruit drinks, cereal grains, cereal products and vegetable oils were analysed for residues of 238 pesticides (289 analytes). National or EU harmonised Maximum Residue Limits (EC-MRLs) were exceeded by 118 samples (5.0 %). Four samples of domestically grown or produced products contained residues above the MRLs.

Cauliflower, peppers, wheat, aubergines, rice, table grapes, cucumbers and peas, in all 505 samples, were analysed in the 2003 EU co-ordinated programme. Thirteen of these samples exceeded the EC-MRLs for the pesticides that were included in the co-ordinated programme.

Residues above the MRL (for baby foods) were found in two out of 101 samples of foods for infants and young children.

A total of 95 enforcement samples of fruits and vegetables were collected and 23 lots (42.3 tons) were prohibited from being sold.

The short-term intake was estimated for all pesticides with an acute referensdose (ARfD) set by WHO. The calculation was based on the residue found in a surveillance (composite) sample and UK consumption data. The ARfD was exceeded for toddlers in ten cases.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

#### Responsibilities

The National Food Administration (NFA) is the responsible authority for the monitoring of pesticide residues in foods.

#### Design of the programmes (priorities, targeting)

The number of samples to be collected of each food is risk related and partly linked to the food's consumption rate and takes into account both the amount of domestic production and the amount of imports from EU-countries and third countries. However, the number is also based on the importance of the foodstuff in the diets of infants and young children as well as residues found in prior samples. The National Food Administration is responsible for the pesticide residue control in Sweden.

#### Sampling: personnel, procedures, sampling points

Samples collected in accordance with the monitoring programme were defined as surveillance samples, i.e. there were no suspicions about excessive amounts of pesticide residues in the lots prior to sampling

Personnel: Plant inspectors from the National Board of Agriculture collected most of the samples in accordance with instructions from NFA.

Procedures: The sampling was done according to Commission Directive 2002/63/EEC. Samples of cereal grains were collected by stream sampling technique. The sample was sealed and labelled with a unique sample identity.

Sampling points: Fresh fruit and vegetables were sampled at wholesalers' warehouses in the first trade channel. The imported cereal grains were sampled at the port where the shipment was discharged. Samples of domestic produced cereal grains were collected at the milling plants. Most of the samples of processed or frozen fruit and vegetables, juices, fruit drinks, rice, cereal products and vegetable oils were collected in retail shops or department stores.

#### Enforcement action

When a surveillance sample contained a pesticide residue above the national or EC-MRL, the National Food Administration prescribed a condition for the offering for sale or other handling of the food or lot to which the food belonged. The remaining part of the lot, if any, was prohibited for



being put on the market. As a follow-up, next lots of the commodity from the grower/exporter were detained and enforcement samples were collected.

### 3. QUALITY ASSURANCE

Status of accreditation of laboratories; number of laboratories

All analyses were carried out at AnalyCen Nordic AB, Lidköping. The laboratory is accredited by the Swedish accreditation authority SWEDAC for all analytical methods used for the NFA's official control of pesticide residues in food of plant origin.

Analytical methods used

Practically all samples of fruit and vegetables were analysed by the multi-residue method. By this method, the samples were extracted with ethyl acetate, cleaned up on an S-X3 gel permeation column and determined by capillary gas chromatography.

In all, by using both multi-residue methods and single residue methods it was possible to determine 238 pesticides corresponding to 289 analytes. About 380 000 residues (pesticide/commodity combinations) were sought (Table A2-Part I-II).

Participation in proficiency tests

The laboratory has participated in one proficiency test organised by EU and in 12 organised by FAPAS, UK (Table G).

Implementation of EU quality control procedures

The EC guidelines SANCO/3103/2000 "Quality Control Procedures for Pesticide Residue Analysis", second edition, 1999/2000 have been implemented as far as practicable.

Analytical uncertainty

**The residue figures found are compared with the MRLs. If the figures, without any correction, are mathematically above the MRL, the sample is defined as an exceeding. However, before any enforcement actions are taken the analytical uncertainty is subtracted from the measured value (95 percent confidence interval). If the corrected figure still exceeds the MRL enforcement actions could be taken.**

### 4. OTHER INFORMATION

Details of homogeneity exercise

In the EU co-ordinated programme the homogeneity factor should be determined for at least certain pesticide/commodity combinations. None of these combinations were found or could be reanalysed.

Background on legislation

Sweden has implemented all EC-MRLs. For some pesticide/commodity combinations National limits are in force.

Details of risk assessment

The short-term intake has been calculated for those pesticides that have an ARfD assigned by WHO. In ten cases the ARfD was exceeded.

## **UNITED KINGDOM**

### **1. SUMMARY OF RESULTS**

Surveys were carried out in support of the Commission recommendation for a harmonised, specific, EU programme. Samples of aubergine, cauliflower, cucumber, grapes, peas (frozen), sweet peppers, wheat grain and rice were analysed for a range of pesticides indicated in the recommendation. Residues were found in 23 of the 72 aubergine samples and 3 contained multiple residues. No MRLs were exceeded. No residues were found in any of the 72 cauliflower samples analysed. Residues were found in 17 of the 73 cucumber samples analysed and 3 contained multiple residues. No MRLs were exceeded. Residues were found in 41 of the 72 grape samples analysed and 25 contained multiple residues. MRLs were exceeded in 4 samples. A residue was found in 1 of the 72 peas (frozen) samples analysed and none contained multiple residues. No MRLs were exceeded. Residues were found in 4 of the 72 sweet pepper samples analysed and none contained multiple residues. No MRLs were exceeded. Residues were found in 54 of the 68 wheat grain samples analysed and 7 contained multiple residues. No MRLs were exceeded. Residues were found in 38 of the 72 rice samples analysed and 7 contained multiple residues. The MRL was exceeded in 1 sample.

Overall, only 0.8% of the samples analysed contained residues exceeding the MRL. This confirmed that in the vast majority of cases pesticides had been applied in accordance with their label requirements, and also provided an assurance to consumers that, where they were being exposed to residues, they were at safe levels. In all cases where MRLs were exceeded, or where there were potential intake issues, a separate consumer risk assessment was carried out to establish whether the level of residues present would lead to an exceedance of the ADI or, where appropriate the ARfD, by a high level consumer. The risk assessments indicated that in the great majority of cases there would be no exceedance. None of the residues found were of concern for human health.

### **2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING**

The UK monitoring programme is overseen by the independent Pesticide Residues Committee (PRC). The purpose of the UK monitoring is threefold: to back up the statutory approvals process for pesticides by checking that no unexpected residues are occurring; to check that residues do not exceed statutory EU and UK maximum residue levels (MRLs) and to check that human dietary intakes of residues are at acceptable levels. This monitoring, together with that carried out by local authorities and the food industry, provides a wide-ranging overview of the residues present in food. In 2003, only 0.8% of samples analysed contained residues above MRLs, and the majority of these would not lead to an exceedance of the acceptable daily intake (ADI).

Samples have generally been obtained at retail level, at fortnightly intervals from two population centres in each of twelve regions of the UK. The centres selected are changed each year. For 2003, we also used Defra officers from both Horticultural Marketing Inspectorate (HMI) and Plant Health & Seeds Inspectorate (PHSI) to collect some samples at non-retail sources; for lettuce, potatoes, apples, peaches and nectarines. This enabled the samples to be more representative of the supply chain. The choice of foodstuffs to be analysed in the programmes generally represents a balance between the levels of consumption of those foodstuffs, information on the possible levels of residues and the need to ensure that as wide a range of commodities as possible is included in the surveillance. Where practicable samples are taken, prepared and analysed according to Commission Directive 2002/63/EC.

In determining its surveillance programme, the PRC considers intelligence data from other sources including publications produced by other UK Agriculture departments. The UK also considers data

published on monitoring carried out by other governments, as well as data and intelligence from industry and other sources.

When the level of pesticide residues found in PRC surveys indicates use of a non-approved pesticide, or the use of a pesticide other than in accordance with the conditions of its approval, the source of the samples involved is investigated to establish the cause of the residues. Further targeted surveillance is normally carried out to check whether the result represented an isolated incident. Where there are persistent or re-occurring problems, then a separate enforcement programme is undertaken. Samples are collected by enforcement officers and follow-up action is taken where any offences have occurred. This may involve legal action and a directed information campaign to assist growers in correct agricultural practice. If problems are identified in retail samples of produce of EU or third country origin, the details are reported to the Member State or exporting country concerned, to allow the relevant authorities to follow up the case. For further details of the 2003 enforcement activity, see: [www.pesticides.gov.uk/fg\\_enforcement.asp?id=1214](http://www.pesticides.gov.uk/fg_enforcement.asp?id=1214)

### 3. QUALITY ASSURANCE

Full details of the PRC's methods of work are contained in *Surveillance: The Estimation of Dietary Exposure to Pesticides - Report of the Working Party on Pesticide Residues: 1991-1993* which is published as Food Surveillance Paper Number 50 available from the Stationery Office (ISBN 0 11 243015 5).

Analytical methods were deemed acceptable if the level of recovery of added pesticide was between 60%-140%, providing that routinely the mean recovery was in the range 70%-110%. Results which exceeded MRLs, or which were unusual in occurrence or were otherwise of particular significance were required to be accompanied by acceptable recovery data and, wherever practicable, to have been confirmed using mass spectrometry. The residues data provided in this report have not been corrected for recovery and are expressed, unless otherwise stated, on the basis of the fresh weight of the sample and as defined by the MRL.

All laboratories carrying out work have taken part in proficiency testing exercises, including the Dutch Chek Monitoring Programme and other international programmes. Recognising the importance of proficiency testing, MAFF (now Defra) started a scheme in 1990 for laboratories known as the Food Analysis Performance Assessment Scheme (FAPAS). All of the laboratories which submit data to the PRC participate in FAPAS. Since 1997, all laboratories have allowed the FAPAS Secretariat to release their individual results and details of their performance to PSD. This allows an independent check of the laboratories' performance to be made. Additionally, all of the laboratories meet the requirements of a recognised accreditation scheme, such as the United Kingdom Accreditation Service (UKAS) or the requirements of Good Laboratory Practice (GLP).

### 4. OTHER INFORMATION

In 2003, the UK carried out the homogeneity exercise on grapes only. The results are reported in Table F.

Due to high levels of methomyl found in samples of Spanish spinach in 2002, the PRC undertook a special survey at the end of 2003. The results were published in the quarter 3 2003 report in March 2004. None of the residues of methomyl found exceeded the MRL.

Due to a number of rapid alert notifications received, the PRC requested early reporting for grapes from India. As a result, MRL exceedances found in some grapes from India, which also eroded the safety margins, were published in advance of the routine publication dates. We also targeted Indian grapes when collecting samples, though no further MRL exceedances were found.

The EU monitoring programme is just part of a larger surveillance programme covering 4,071 samples. Full results are published quarterly and can be found on the PRC website at [www.prc-uk.org](http://www.prc-uk.org).

## **NORWAY**

### **1. SUMMARY OF RESULTS**

A total of 2159 samples of fresh and frozen fruit, vegetables and cereals, both imported (66 %) and domestically produced (34 %) were analysed in the monitoring programme for 2003. In addition the monitoring programme included 171 samples of processed products and a few follow-up samples.

A total of 114 different commodities were analysed. The monitoring programme covered 206 pesticides including some isomers and breakdown products.

Of the samples of fresh fruit and vegetables, including potatoes, 63 % were without detectable pesticide residues. The MRLs were exceeded in 2.2 % of the samples (0.7 % in domestic and 3.0 % in imported samples). Among the domestic products violations occurred most frequently in a variety of herbs from the same farmer. Among the imported products strawberries, carambola, passion fruit, mango, beans with pod, papaya and spring onion had the highest number of samples exceeding the MRL.

Analysis of 85 samples of baby food showed that none exceeded the MRL.

In a project on fruit and vegetables imported directly to retail from third countries 95 samples were analysed. Pesticide residues were found in 12.6 % of the samples, 4.2 % of the samples had residues that exceeded the MRL.

In a project on herbal tea 86 samples were analysed. Pesticide residues were found in one sample. The sample did not exceed the MRL.

Of a total of 97 samples of cereals 68 had no detectable pesticide residues. No samples exceeded the MRL.

57 samples of organic grown products were analysed. Pesticide residues were not found in any samples.

### **2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING**

The samples were mainly taken at wholesaler's warehouse in different parts of Norway. Some samples were taken at retailers, farms or at market places. The number of samples of each commodity does not reflect their share of the market as more samples were taken of commodities suspected to contain residues. Trained inspectors from the municipal food control authorities were responsible for taking samples in accordance with a national sample plan and official guidelines for sampling.

For some of the samples found to contain residues exceeding the MRL, compliance samples were collected as a follow-up. The consignments were suspended until the results of the analyses were available.

### **3. QUALITY ASSURANCE**

The Norwegian Crop Research Institute, Pesticide Laboratory, was responsible for the analyses. The Pesticide Laboratory was accredited on April 1st 1997. The accreditation is according to NS-EN ISO/IEC 17025. The analyses were carried out in accordance with the Quality Assurance Manual of the Pesticide Laboratory and the EU Guidelines for Pesticide Residues Analysis. As a part of the quality assurance programme the laboratory regularly participates in international intercalibrations / proficiency tests.

All samples of fruit and vegetables were analysed using a GC multi-residue method, based on extraction with acetone (Luke method), covering 153 pesticides, isomers and breakdown products. Ten pesticides were analysed by LC/MS using the same extraction method. LC/MS was also used for analysis of N-methylcarbamates, chlormequat/mepiquat, diquat, dithianon, propamocarb and

dinocap. Biphenyl, orto-Phenylphenol, progargite and isoproturon were analysed using GC/MS. Phenoxyacid herbicides and glyphosate/AMPA were analysed by GC/MS after derivatization. The dithiocarbamates were determined with a spectrophotometric method. The reporting levels are the limit of determination for all methods.

When a pesticide residue exceeds the MRL, quantitative determination is carried out on three replicate samples using three-level calibration, recovery is checked and the identity of the pesticide confirmed by GC/MS or LC/MS. The pesticide residues figures found are then compared to the MRLs. If the figures, un-corrected, are mathematically above the MRL, the sample is defined as an exceeding. However, before any enforcement action is taken the predicted analytical uncertainty is subtracted from the measured value (95 % confidence interval). If this uncertainty-corrected figure still exceeds the MRL enforcement action is taken.

## ICELAND

### 1. SUMMARY OF RESULTS

This report summarizes the results of the monitoring of pesticide residues in fruits, vegetables and cereals in Iceland. A total of 315 samples were taken in the year 2003, approximately 24% of domestic commodities, 76% of imported commodities, there of 33% imported commodities from third countries. 300 samples were taken of fresh fruits and vegetables and 15 samples of cereals. 59% of the samples of fruit and vegetables were without detectable residues, 40% were with residues at or below MRL and 1% had residues, which exceeded the MRL. No residues were detected in the samples of cereals taken. Commodities originated from 28 countries, most of them came from The Netherlands and Spain.

As a main rule the maximum residue limits (MRL's) conform to EU directives, but in few cases, where EU has not set any limits, they are in accordance with either CODEX Alimentarius or regulations from other Nordic countries. When a pesticide residue exceeds MRL, a new sample is analysed to confirm the results. No actions are taken unless the value exceeds the MRL plus a standard deviation.

Iceland only participated partly in the co-ordinated program.

### 2. ORGANISATION OF MONITORING PROGRAMMES AND SAMPLING

- Responsibilities and design of programmes: Sampling plan is made every year based on information on import volumes and domestic production. Experience is also taken into account, as to what pesticide residues are most often analysed in a particular product.
- Sampling: Samples were collected according to national regulation no 284/2002 on contaminants in foodstuffs which is based on EC directives. The samples are taken at wholesaler's warehouses in Reykjavik. The Environmental health and protection office collects the samples. Chemical analysis of fruit and vegetables was done at the laboratory of the Environment and food agency of Iceland. Analysis of cereals was done at **AnalyCen Nordic** laboratory in Sweden. The most frequently found residues were: thiabendazol, imazalil, orthophenylphenol, chlorpyrifos, diphenylamine, prochloraz, iprodione, dicofol and metidation.
- Enforcement action: Actions are taken if the pesticide residues are over MRL plus deviation.

### 3. QUALITY ASSURANCE

- Status of accreditation of laboratories and number of laboratories: Two laboratories were used. The Laboratory of the Environment and food agency of Iceland, which has not yet been accredited and **AnalyCen Nordic**, which is accredited.
- Analytical methods used: Extraction with organic solvents followed by GC-MS analysis.
- Participation in proficiency tests: The laboratory of the Environmental and Food Agency takes part in FAPAS.
- Implementation of EU quality control procedures: The following parts of EU quality control procedures were followed: Sampling, transport, processing and storage of samples, pesticide, standards, calibration, solution, extraction and concentration, contamination and inference, analytical calibration and chromatographic integration, analytical methods and analytical performance and proficiency testing and analysis of reference material. Analytical uncertainty estimation is applied on results.

## LIECHTENSTEIN

### *1. Summary of Results*

This report summarizes the results of the co-ordinated and the national pesticide monitoring programme in fruits, vegetables and cereals on the Liechtenstein market in 2003. This report has been prepared according to the recommendation of the ESA as far as technically possible.

A total of 48 samples were analysed out of which 4 fruits, 36 vegetables and 8 cereals.

In 25 samples no pesticide residues were detected. 13 samples showed pesticide contamination at or below national and EC MRLs. One sample contained a residue level higher than the EC-MRL, determined for a special product-pesticide residues combination concerned.

The total number of residues analyzed for is 41. The most frequently found residues derived from Dithiocarbamat-/Thiuramdisulfid-fungicides, pesticides of the Maneb-group. 5 different kind of vegetables showed contamination with this pesticide.

Summary of results:

	Total nr. of samples analysed	Nr. of samples without residues	Nr. of samples with residues at or below the MRL	Nr. of exceedings of EC-MRLs	Nr. of exceedings of N-MRLs
Co-ordinated and national programme, vegetables and cereals	48	25	13	1	-

### *2. Organisation of Monitoring Programme and Sampling*

The samples were collected according to the annual sampling plan prepared by the Office of Food Inspection and Veterinary Affairs. The sampling plan is based on the domestic production situation in the agricultural sector and on the ESA co-ordinated monitoring programme. The programme started in spring 2003.

Samples of fresh fruits, vegetables and cereals were collected mostly from retailers, but also from food processing plants and 8 samples from farm gates. Samples were taken by trained staff-members of the OFV, mostly according to directive 2002/63/EC. The size of a laboratory sample was at least 1 kg.

### *3. Quality Assurance*

Analyses have been carried out to 100 % in the Labor Dr. Matt AG, Im alten Riet 36, FL-9494 Schaan, a private laboratory, with which the OFV has a contract defining the co-operation, the tasks and the obligations. The laboratory is accredited in accordance with the provisions of article 3 of directive 93/99 (according to DIN EN ISO/IEC 17025 [see copy of accreditation certificate in the annex]).

All samples were analysed by a gas chromatographic multiresidue method (modified German official method DFG S19). This method consists of an acetone/cyclohexane/ethylacetate extraction and a clean-up on a bio beads S-X3 gel permeation column. The extract was analysed by GC with selective detectors (ECD, NPD and FPD) / exception: Carbendazim and Thiabendazol were analysed by HPLC. When a pesticide residue exceeded the reporting level, the identity of the pesticide was confirmed by GC-MS.



Dithiocarbamates were also determined on each sample by using an CS<sub>2</sub> evolution method. The CS<sub>2</sub> is determined by headspace analysing using GC with ECD (DIN 12396-2).

The laboratory carrying out the analyses participates as a part of its quality assurance programme in proficiency tests.

The analytical results are governed by a quality assurance system. The laboratory takes into account, as far as it is possible for a laboratory of its size, of the document "Quality control Procedures for Pesticide Residue Analyses"- as set out in Annex II to the Recommendation of the ESA for the year 1999, OJ L 74, 23.3.2000.

The reporting levels are the same as the limits of quantification (generally in the range of 0,005 - 0,05 mg/kg). The limits of quantification are determined by recovery tests.

Analytical uncertainty applied on results varies depending to substance and method between 10 – 15 %.

Schaan, 22 July 2004 map/tos

Encl.: Tables A - G